PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 15 May 2001 (15.05.01)	
International application No. PCT/SE00/01670	**Applicant's or agent's file reference** P11140/KBN:GT
International filing date (day/month/year) 31 August 2000 (31.08.00)	**Priority date** (day/month/year) 01 September 1999 (01.09.99)
Applicant FINGAL, Lars et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

30 March 2001 (30.03.01)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland**	Authorized officer Athina Nickitas-Etienne
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 00/01670

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D04H 1/46
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3485706 A (F.J. EVANS), 23 December 1969 (23.12.69) --	1-17
Y	DE 4013946 A1 (HOECHST AG), 31 October 1991 (31.10.91) --	1-10
Y	US 5286553 A (K. HARAGUCHI ET AL), 15 February 1994 (15.02.94) --	11-17

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
24 November 2000	03-01-2001
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Christer Wendenius / MRo Telephone No. +46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 1998)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/SE 00/01670

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3485706 A	23/12/69	NONE	
DE 4013946 A1	31/10/91	AT 107974 T	15/07/94
		DE 59102054 D	00/00/00
		DK 455193 T	07/11/94
		EP 0455193 A,B	06/11/91
		SE 0455193 T3	
		ES 2057651 T	16/10/94
		IE 65104 B	04/10/95
		JP 4228641 A	18/08/92
		PT 97516 A	30/07/93
		US 5293676 A	15/03/94
		US 5424123 A	13/06/95
US 5286553 A	15/02/94	CA 2010559 A,C	21/08/91
		DE 68923964 D,T	15/02/96
		EP 0409993 A,B	30/01/91
		JP 2097812 C	02/10/96
		JP 3047713 A	28/02/91
		JP 8009164 B	31/01/96
		WO 9007024 A	28/06/90

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

AKERMAN, Marten
Albihns Malmö AB
P.O. Box 4289
S-203 14 Malmö
SUEDE

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing *(day/month/year)*	30.10.2001
Applicant's or agent's file reference P11140/KBN:GT	**IMPORTANT NOTIFICATION**

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/SE00/01670 ✓	31/08/2000	01/09/1999 ✓

Applicant
SCA HYGIENE PRODUCTS AB et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Abadie, N Tel.+49 89 2399-2746

Form PCT/IPEA/416 (July 1992)

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
P11140/KBN:GT		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/SE00/01670	31/08/2000	01/09/1999
International Patent Classification (IPC) or national classification and IPC D04H1/46		
Applicant SCA HYGIENE PRODUCTS AB et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
30/03/2001	30.10.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Lanniel, G Telephone No. +49 89 2399 2062

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/SE00/01670

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:
Description, pages:

1-17	as originally filed	

Claims, No.:

17	as originally filed	
1-16	with telefax of	17/10/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   - ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
   - ☐ the language of publication of the international application (under Rule 48.3(b)).
   - ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   - ☐ contained in the international application in written form.
   - ☐ filed together with the international application in computer readable form.
   - ☐ furnished subsequently to this Authority in written form.
   - ☐ furnished subsequently to this Authority in computer readable form.
   - ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   - ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   - ☐ the description, pages:
   - ☐ the claims, Nos.:
   - ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims 1-16
	No:	Claims
Inventive step (IS)	Yes:	Claims 1-16
	No:	Claims
Industrial applicability (IA)	Yes:	Claims 1-16
	No:	Claims

2. Citations and explanations
   see separate sheet

The invention relates to a method for manufacturing a nonwoven by hydroentangling where the flexural rigidity of the fiber components used does not limit the degree of entangling, and consuming less energy. The invention relates to the nonwoven fabric thus produced.

This result is achieved by raising the temperature at the very moment of entangling, so it is possible to reduce the flexural rigidity of the fibers and achieve a higher degree of entangling. During the hydroentanglement, the polymer fiber is imparted a temperature which is equal to or exceeds the glass transition temperature for the polymer fiber and below its melting point.

US-A-3 485 706 discloses a method for hydroentangling polymer fibers. There is no disclosure that the polymer fibers to be hydroentangled are heated to a temperature between the glass transition temperature and the melting point.In order to cause fibers to undergo shrinkage, there is a treatment with hot water but after the hydroentanglement step.

10/069873
JC Rec'd PCT/PTO 01 MAR 2002

CLAIMS

1. Method of hydroentangling polymer fibers to manufacture a nonwoven fabric, **characterized in** that the polymer fiber, at the moment of hydroentangling, is imparted a temperature equal to or exceeding the glass transition temperature (Tg) of the polymer fiber and being less than the melting point of the polymer fiber.

2. Method according to claim 1, **characterized in** that the polymer fiber has an initial modulus ≥ 50 cN/tex, at room temperature.

3. Method according to claim 1, **characterized in** that the polymer fiber has an initial modulus ≥ 100 cN/tex, at room temperature.

4. Method according to claim 3, **characterized in** that the polymer fiber has an initial modulus of 100 – 2000 cN/tex, especially 500 – 1500 cN/tex, more particularly 200 – 750 cN/tex, and even more particularly 250 – 600 cN/tex, at room temperature.

5. Method according to any of claims 1 – 4, **characterized in** that the temperature is achieved with the aid of hot or superheated water.

6. Method according to any of claims 1 – 4, **characterized in** that the temperature is achieved with the aid of IR-heat.

7. Method according to any of claims 1 – 4, **characterized in** that the temperature is achieved with the aid of microwaves.

8. Method according to any of claims 1 – 7, **characterized in** that the polymer fiber has a glass transition temperature (Tg) of ≥ 20°C.

9. Method according to any of claims 1 – 8, **characterized in** that the polymer fiber has a glass transition temperature (Tg) of 20 - 100°C, especially 50 - 70°C.

10. Method according to any of claims 1 – 9, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid , polyamide or polypropylene, or copolymers or mixtures thereof.

11. Hydroentangled nonwoven fabric obtainable by the method of any of claims 1-10 comprising polymer fibers, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 20 - 100°C and an initial modulus of 200 – 750 cN/tex at room temperature.

12. Nonwoven fabric according to Claim 11, **characterized in** that the polymer fibers in the nonwoven fabric have an initial modulus of 250 – 600 cN/tex at room temperature.

13. Nonwoven fabric according to any of claims 11-12, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 50 - 70°C.

14. Nonwoven fabric according to any of claims 11 – 13, **characterized in** that the nonwoven fabric has a bulk specific volume of ≥ 8 cm^3/g.

15. Nonwoven fabric according to claim 14, **characterized in** that the nonwoven fabric has a bulk specific volume of 8 – 15 cm^3/g, especially 10 – 15 cm^3/g.

16. Nonwoven fabric according to any of claims 11– 15, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid , poly-amide or polypropylene, or copolymers or mixtures thereof.

RECORD COPY

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No. PCT/ SE 00 / 0 1 6 7 0

International Filing Date 2000 -08- 31

The Swedish Patent Office
PCT International Application

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference *(if desired) (12 characters maximum)* P11140/KBN:GT

Box No. I TITLE OF INVENTION

FIBER BONDING

Box No. II APPLICANT

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

SCA HYGIENE PRODUCTS AB
S-405 03 GÖTEBORG
Sweden

RO/SE

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State *(that is, country)* of nationality: Sweden

State *(that is, country)* of residence: Sweden

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

FINGAL, Lars
Uddevallaplatsen 12
SE-416 79 GÖTEBORG
Sweden

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: Sweden

State *(that is, country)* of residence: Sweden

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below ~~is hereby~~ has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

See Supplemental Sheet (No. 3)

Telephone No. +46 40 705 40

Facsimile No. +46 40 611 96 89

Teleprinter No.

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

STRÅLIN, Anders
Andalen 95
SE-423 38 TORSLANDA
Sweden

This person is:
- [ ] applicant only
- [x] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality: Sweden

State *(that is, country)* of residence: Sweden

This person is applicant for the purposes of:
- [ ] all designated States
- [ ] all designated States except the United States of America
- [x] the United States of America only
- [ ] the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- [ ] applicant only
- [ ] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of:
- [ ] all designated States
- [ ] all designated States except the United States of America
- [ ] the United States of America only
- [ ] the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- [ ] applicant only
- [ ] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of:
- [ ] all designated States
- [ ] all designated States except the United States of America
- [ ] the United States of America only
- [ ] the States indicated in the Supplemental Box

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

This person is:
- [ ] applicant only
- [ ] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

This person is applicant for the purposes of:
- [ ] all designated States
- [ ] all designated States except the United States of America
- [ ] the United States of America only
- [ ] the States indicated in the Supplemental Box

- [ ] Further applicants and/or (further) inventors are indicated on another continuation sheet.

Supplemental Box *If the Supplemental Box is not used, this sheet should not be included in the request.*

*1. If, in any of the Boxes, **the space is insufficient** to furnish all the information: in such case, write "Continuation of Box No. ..." [indicate the number of the Box] and furnish the information in the same manner as required according to the captions of the Box in which the space was insufficient, in particular:*

*(i) **if more than two persons are involved as applicants and/or inventors** and no "continuation sheet" is available: in such case, write "Continuation of Box No. III" and indicate for each additional person the same type of information as required in Box No. III: The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below;*

*(ii) if, in Box No. II or in any of the sub-boxes of Box No. III, the indication **"the States indicated in the Supplemental Box"** is checked: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the applicant(s) involved and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is applicant;*

*(iii) if, in Box No. II or in any of the sub-boxes of Box No. III, **the inventor or the inventor/applicant is not inventor for the purposes of all designated States or for the purposes of the United States of America**: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the inventor(s) and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is inventor;*

*(iv) if, in addition to the agent(s) indicated in Box No. IV, there are **further agents**: in such case, write "Continuation of Box No. IV" and indicate for each further agent the same type of information as required in Box No. IV;*

*(v) if, in Box No. V, the name of any State (or OAPI) is accompanied by the indication "**patent of addition**," or "**certificate of addition**," or if, in Box No. V, the name of the United States of America is accompanied by an indication "**continuation**" or "**continuation-in-part**": in such case, write "Continuation of Box No. V" and the name of each State involved (or OAPI), and after the name of each such State (or OAPI), the number of the parent title or parent application and the date of grant of the parent title or filing of the parent application;*

*(vi) if, in Box No. VI, there are **more than three earlier applications whose priority is claimed**: in such case, write "Continuation of Box No. VI" and indicate for each additional earlier application the same type of information as required in Box No. VI;*

*(vii) if, in Box No. VI, **the earlier application is an ARIPO application**: in such case, write "Continuation of Box No. VI", specify the number of the item corresponding to that earlier application and indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed.*

*2. If, with regard to the **precautionary designation statement** contained in Box No. V, the applicant wishes to exclude any State(s) from the scope of that statement: in such case, write "Designation(s) excluded from precautionary designation statement" and indicate the name or two-letter code of each State so excluded.*

*3. If the applicant claims, in respect of any designated Office, the benefits of provisions of the national law concerning **non-prejudicial disclosures or exceptions to lack of novelty**: in such case, write "Statement concerning non-prejudicial disclosures or exceptions to lack of novelty" and furnish that statement below.*

Continuation of Box No. IV

ÅKERMAN, Mårten;
KITZLER, Michael
LINDBERG, Olle
BERGMAN, Kerstin
MANNERLÖF TENNING, Marie
DANFELTER, Maria
LINDAHL, Dan
RÜTER, Viveca
c/o Albihns Patentbyrå Malmö AB
P.O. Box 4289
SE-203 14 MALMÖ
Sweden

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) *(mark the applicable check-boxes; at least one must be marked)*:

Regional Patent

☒ **AP** **ARIPO Patent:** **GH** Ghana, **GM** Gambia, **KE** Kenya, **LS** Lesotho, **MW** Malawi, **MZ** Mozambique, **SD** Sudan, **SL** Sierra Leone, **SZ** Swaziland, **TZ** United Republic of Tanzania, **UG** Uganda, **ZW** Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT

☒ **EA** **Eurasian Patent:** **AM** Armenia, **AZ** Azerbaijan, **BY** Belarus, **KG** Kyrgyzstan, **KZ** Kazakhstan, **MD** Republic of Moldova, **RU** Russian Federation, **TJ** Tajikistan, **TM** Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT

☒ **EP** **European Patent:** **AT** Austria, **BE** Belgium, **CH and LI** Switzerland and Liechtenstein, **CY** Cyprus, **DE** Germany, **DK** Denmark, **ES** Spain, **FI** Finland, **FR** France, **GB** United Kingdom, **GR** Greece, **IE** Ireland, **IT** Italy, **LU** Luxembourg, **MC** Monaco, **NL** Netherlands, **PT** Portugal, **SE** Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT

☒ **OA** **OAPI Patent:** **BF** Burkina Faso, **BJ** Benin, **CF** Central African Republic, **CG** Congo, **CI** Côte d'Ivoire, **CM** Cameroon, **GA** Gabon, **GN** Guinea, **GW** Guinea-Bissau, **ML** Mali, **MR** Mauritania, **NE** Niger, **SN** Senegal, **TD** Chad, **TG** Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT *(if other kind of protection or treatment desired, specify on dotted line)* .....

National Patent *(if other kind of protection or treatment desired, specify on dotted line)*:

☒ **AE** United Arab Emirates
☒ **AG** Antigua and Barbuda
☒ **AL** Albania .....
☒ **AM** Armenia .....
☒ **AT** Austria .....
☒ **AU** Australia .....
☒ **AZ** Azerbaijan
☒ **BA** Bosnia and Herzegovina .....
☒ **BB** Barbados
☒ **BG** Bulgaria .....
☒ **BR** Brazil .....
☒ **BY** Belarus .....
☒ **BZ** Belize
☒ **CA** Canada
☒ **CH and LI** Switzerland and Liechtenstein
☒ **CN** China .....
☒ **CR** Costa Rica .....
☒ **CU** Cuba .....
☒ **CZ** Czech Republic .....
☒ **DE** Germany .....
☒ **DK** Denmark .....
☒ **DM** Dominica
☒ **DZ** Algeria .....
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☒ **GH** Ghana .....
☒ **GM** Gambia
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☒ **HU** Hungary .....
☒ **ID** Indonesia
☒ **IL** Israel .....
☒ **IN** India .....
☒ **IS** Iceland
☒ **JP** Japan .....
☒ **KE** Kenya .....
☒ **KG** Kyrgyzstan .....
☒ **KP** Democratic People's Republic of Korea .....
☒ **KR** Republic of Korea .....
☒ **KZ** Kazakhstan .....
☒ **LC** Saint Lucia
☒ **LK** Sri Lanka
☒ **LR** Liberia
☒ **LS** Lesotho .....
☒ **LT** Lithuania
☒ **LU** Luxembourg
☒ **LV** Latvia
☒ **MA** Morocco .....
☒ **MD** Republic of Moldova .....
☒ **MG** Madagascar .....
☒ **MK** The former Yugoslav Republic of Macedonia .....
☒ **MN** Mongolia
☒ **MW** Malawi .....
☒ **MX** Mexico .....
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☒ **TR** Turkey .....
☒ **TT** Trinidad and Tobago .....
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☒ **UA** Ukraine .....
☒ **UG** Uganda .....
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Check-box reserved for designating States which have become party to the PCT after issuance of this sheet:

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Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. *(Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)*

Box No. VI PRIORITY CLAIM ☐ Further priority claims are indicated in the Supplemental Box.

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is: national application: country	regional application:* regional Office	international application: receiving Office
item (1) 1 September 1999	9903075-1	Sweden (SE)		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office)* identified above as item(s): 1

** Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.*

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used)* : ISA / SE

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority)*:

Date (day/month/year)	Number	Country (or regional Office)
1 September 1999 ~~1 September 1999~~	9903075-1 SE99/01173	Sweden SE

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following **number of sheets**:

request	:	4 5
description (excluding sequence listing part)	:	12
claims	:	2
abstract	:	1
drawings	:	
sequence listing part of description	:	
Total number of sheets	:	19

This international application is **accompanied by** the item(s) marked below:

1. ☒ fee calculation sheet
2. ☒ separate signed power of attorneys
3. ☐ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
8. ☐ nucleotide and/or amino acid sequence listing in computer readable form
9. ☐ other *(specify)*:

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: Swedish

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Malmö, this 30th day of August 2000

Kerstin Bergman

Kerstin Bergman, agent

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1. Date of actual receipt of the purported international application:	2000 -08- 31	2. Drawings: ☐ received: ☒ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): ISA / SE	6. ☐ Transmittal of search copy delayed until search fee is paid.	

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Date of receipt of the record copy by the International Bureau: 25 SEPTEMBER 2000 ~~(25. 09. 00)~~

FIBERBINDNING

TEKNISKT OMRÅ

Uppfinninge ymerfibrer till ett fibertyg samt fiberty

Enligt uppfi lroentangling, varvid fibrerna i hydroentar verstiger polymerfiberns glasomvandli fiberns smälttemperatur.

UPPFINNINGENS I

Att tillverka tyger och vävar är känt sedan lång tid tillbaka. Förutom de välkända metoderna, vävning, stickning och virkning finns numera ett stort antal metoder för framställning av fibertyg eller s.k. non-wovenmaterial. Sådana material kan tillverkas av såväl syntetiska fibrer som naturfibrer. En del metoder utnyttjar hög värme för att smälta samman fibrerna, s. k. termobindning.

Det finns också andra bindningsmetoder som t. ex. "stitch-bonding" och hydroentangling eller spunlacing. Hydroentangling eller spunlacing är en teknik som introducerades på 1970-talet. Metoden innebär att en fiberbana bildas, antingen en torrlagd sådan eller en våtlagd, varefter fibrerna entanglas, dvs. trasslas samman med hjälp av mycket fina vattenstrålar under högt tryck. Flera rader av vattenstrålar riktas mot fiberbanan som uppbärs av en rörlig vira eller trumma. Avslutningsvis torkas den entanglade fiberbanan.

I hydroentanglade material används många olika typer och blandningar av fibrer, som t. ex. syntetiska stapelfibrer, syntetiska kontinuerliga fibrer, stapelfibrer av regenererad cellulosa samt massafibrer. Exempel på kommersiellt tillgängliga fibrer av regenererad cellulosa är rayon, viskos och lyocell.

Hydroentangling är ett sätt att binda fibrer utan att bindemedel eller bindefibrer behövs. Hydroentanglat material eller spunlace-material av hög kvalitet kan framställas till en rimlig kostnad och därvid till ett material som uppvisar hög absorptionsförmåga, har goda mekaniska egenskaper och har en hög textil komfort. De användes bl. a. som avtorkningsmaterial for hushållsbruk eller industriellt bruk, som engångsmaterial inom sjukvård och för hygienbruk, etc.

En förutsättning för att en sammanvävd fiberbana skall kunna bildas med hjälp av hydroentangling är att de ingående fibrer som skall entanglas har för ändamålet rätt egenskaper.

En av flera kritiska faktorer är fiberns böjstyvhet = E x I, där E är fiberns E-modul eller elasticitetsmodul och I är tröghetsmomentet. Tröghetsmomentet är en

sektionsstorhet (I=πd4/64 för cirkulärt tvärsnitt) som beror av fiberns diameter. E-modulen är således en materialparameter och denna är temperaturberoende.

En styv fiber är svårare att entangla och kräver mer specifik energi (kWh/ton) för att låta sig bindas än en mjuk fiber, vilket i sin tur begränsar utbudet av fiberslag som är tekniskt och kommersiellt intressanta att använda för denna teknologi.

Fibrer gjorda av termoplastiska polymerer som t.ex. polypropen, polyester, polyamid är vanligt förekommande vid hydroentangling.

Egenskaperna hos termoplastiska och andra syntetiska fibrer är beroende av egenskaperna hos den eller de ingående polymererna och den utnyttjade processens beskaffenheter. Ofta kan polymerens egenskaper inte utnyttjas fullt ut, utan en kompromiss måste göras av processtekniska skäl.

Det är typiskt mycket svårt att framställa en fiber som är mycket stark och samtidigt har en låg E-modul. Fiberns styrka avgörs till stor del av molekylkedjornas orientering, längd och inbördes attraktionskraft. Fiberns hållfasthet och E-modul följer härvid samma tendens på så sätt att en hög fiberstyrka samtidigt innebär en hög E-modul.

I CA 841.938 beskrives framställning av ett fibertyg genom hydroentangling varvid vatten under högt tryck pressas genom en hålförsedd bärare och mot ett ark av en fibersuspension för att ge upphov till en intrassling av fibrerna.

WO 95/06769 beskriver en metod och en apparat för att åstadkomma en smältbindning och ev. en entangling av fibrer i en fiberväv, t. ex. ett fibertyg eller s.k. non-woven. Härvid utnyttjas en ångstråle eller en stråle överhettad ånga för att såväl smälta som entangla fibrerna. I det fall sådana strålar utnyttjas som normalt tillämpas vid hydroentangling så måste de vara tillräckligt varma för att smälta en smältkomponent som har införlivats i fiberväven. WO 95/06769 beskriver således ett förfarande, varvid en viss smältning av en ingående smältkomponent alltid uppnås. Denna smältkomponent kan antingen vara själva fibrerna eller en tillsatt smältkomponent i form av pulver eller granulat. Något förfarande för renodlad hydroentangling beskrivs ej.

US 3,322,584 beskriver ett smältbindningsförfarande för sammanbindning av två plastvävar. Den beskrivna metoden kan också utnyttjas för att sammanbinda två lager plastfibrer, men återigen avses en smältbindning och således är den utnyttjade temperaturen tillräckligt hög för att smälta trådarna.

US 5,069,735 beskriver en metod för kantsmältning av avgränsade dukar eller vävar i syfte att lösa problemet med att dessa dukar normalt flockar av sig och är olämpliga att användas t. ex. vid operationer.

US 3,192,560 beskriver ett förfarande för att kontrollerat smältbinda fibertrådar med ett lämpligt medium, t. ex. ånga eller överhettad ånga, varvid temperaturen hålles kring eller strax under smälttemperaturen för fibertrådarna.

Ett problem med hydroentangling är att de ingående fiberkomponenterna måste ha en böjstyvhet sådan att fibrerna låter sig entanglas inom rimliga energinivåer. Detta innebär en begränsning vad avser användbara fiberslag och medför att tunna fibrer eller fibrer med låg E-modul måste användas, även om fibrerna i sig inte är optimala för fiberflorets formering eller för det färdiga materialets funktionella egenskaper.

Ett ändamål med föreliggande uppfinning är att åstadkomma ett förfarande för framställning av ett fibertyg genom hydroentangling, där ingående fiberkomponenters böjstyvhet inte i lika hög grad som tidigare innebär en begränsning för entanglingsgraden.

Ett ytterligare ändamål med föreliggande uppfinning är att åstadkomma ett förfarande som ger en potential jämfört med dagens metoder vilken t. ex. kan utnyttjas till att tillverka fibertyg med grova fibrer, ge lägre energiförbrukning eller ge ett starkare tyg.

Ännu ett ändamål med föreliggande uppfinning är att tillhandahålla ett fibertyg med speciella egenskaper, såsom goda mekaniska egenskaper, hög bulk, m.m.

SAMMANFATTNING AV UPPFINNINGEN

Vi har nu funnit att vi genom att åstadkomma en temperaturhöjning i själva entanglingsögonblicket, kan sänka fibrernas böjstyvhet och uppnå en högre grad av intrassling.

Vi har vidare funnit att det enbart är i själva entanglingsögonblicket som en alltför hög E-modul är en nackdel och genom att sänka E-modulen just vid själva hydroentanglingen för att därefter låta E-modulen återgå till den ursprungliga nivån, åstadkommes ett sätt och ett material som har stora fördelar jämfört med tidigare kända.

Därvid minskar beroendet av den begränsning det innebär att i fibertillverkningsprocessen kompromissa mellan egenskaperna hållfasthet och styvhet. Fiberns hållfasthet kan i stället optimeras fullt ut.

Samtidigt ges möjlighet att välja fibrer till det fibertyg som skall bindas utifrån andra kriterier än den begränsning som entanglingsprocessen innebär. I många fall är det en fördel med styva fibrer i det färdigbundna fibertyget beroende på vad materialet skall användas till.

Fibrer som redan är relativt väl anpassade för hydroentangling kan optimeras ytterligare, vilket innebär förbättrade materialegenskaper och/eller lägre energiförbrukning i processen.

Enligt uppfinningen åstadkommes ett förfarande för hydroentangling av polymerfibrer för framställning av ett fibertyg. Polymerfibern ges, vid hydroentanglingsögonblicket, en temperatur som är lika med eller överstiger glasomvandlingstemperaturen för polymerfibern samt understiger smälttemperaturen för polymerfibern.

Enligt uppfinningen åstadkommes vidare ett hydroentanglat fibertyg innefattande polymerfibrer, varvid polymerfibrerna i fibertyget har en E-modul ≥ 50 cN/tex.

Vidare åstadkommes enligt uppfinningen ett hydroentanglat polymerfibertyg med en bulkmassa av ≥ 8 cm^3/g.

Ytterligare utföringsformer framgår av de medföljande underkraven.

DETALJERAD BESKRIVNING AV UPPFINNINGEN

Enligt uppfinningen värmes polymerfibern så att den vid entanglingsögonblicket når en temperatur över polymerfiberns glasomvandlingstemperatur (Tg). Vid denna temperatur ökar rörligheten i molekylerna i sådan grad att styvheten påverkas dramatiskt och en sänkning av elasticitetsmodulen eller E-modulen upp till flera 10-potenser kan erhållas.

Syntetiska polymerers mekaniska egenskaper förändras dramatiskt vid polymerens glasomvandlingstemperatur. Genom att momentant värma den önskade fibern till glasomvandlingstemperaturen eller strax däröver vid hydroentanglingen sänks fiberns böjstyvhet och intrasslingsgraden eller entanglingsgraden i fibertyget ökas.

Många olika typer och blandningar av polymerfibrer kan utnyttjas. Speciellt föredraget är, enligt uppfinningen, ett fibertyg som helt eller delvis innefattar syntetiska polymerfibrer, eller blandningar eller sampolymerer av syntetiska polymerfibrer. Beroende framförallt på ändamålet med fibertyget väljs typ av fiber och inblandningsgraden av naturfibrer. Ju mer andel syntetisk polymer som får ingå i fibertyget, desto större är möjligheterna.

Exempel på fibrer som kan användas i materialet enligt föreliggande uppfinning är syntetiska stapelfibrer, syntetiska kontinuerliga fibrer, stapelfibrer av regenererad cellulosa, naturfibrer såsom växtfibrer, massafibrer eller blandningar av dessa. Exempel på kommersiellt tillgängliga fibrer av regenererad cellulosa är rayon, viskos och lyocell. Exempel på syntetiska fibrer är fibrer av polyester, polylaktid, polyamid, polypropen, polybutylentereftalat (PBT), polyeten (PE), polyetylentereftalat (PET) och sampolymerer därav, såsom polyesteramider.

Bikomponentfibrer, dvs. fibrer med en kärna av en första polymer, t.ex. PET och ett hölje av en andra polymer,t.ex. PE kan användas. De syntetiska polymerfibrerna kan innefatta dels polymerfibrer tillverkade av naturfibrer dels polymerfibrer tillverkade av syntetiska fibrer. Även kontinuerliga filament, som t. ex. meltblown och spun bond fibrer kan användas och vidare kan profilerade s.k. kapillärfibrer utnyttjas. Dessa profilerade fibrer är ofta mycket styva och normalt svåra att hantera, men kan entanglas med hjälp av föreliggande förfarande. Även blandningar av dessa olika fibrer kan utnyttjas. En typisk blandning är 40-50 % långa, syntetiska fibrer och resten cellulosa, men alla blandningar är tillämpningsbara. Massafibrerna kan vara av kemisk, mekanisk, termomekanisk, kemimekanisk eller kemitermomekanisk massa (CTMP). Inblandning av mekaniska, termomekaniska, kemimekaniska eller kemitermomekaniska massafibrer ger ett material med högre bulk och förbättrad absorption och mjukhet, vilket beskrivs i SE 9500585-6.

Enligt uppfinningen utnyttjas framförallt termoplastiska, syntetiska polymerer och däribland speciellt semi-kristallina polymerer. Även amorfa polymerer kan utnyttjas.

Uppvärmningen av polymerfibern i hydroentanglingsögonblicket kan göras på mångahanda sätt. Ett sätt att åstadkomma en momentan temperaturhöjning i processen är att värma entanglingsvattnet till en temperatur så att fibern, i själva hydroentanglingsögonblicket, når en temperatur över Tg. Lämpligen utnyttjas detta förfarande då polymerens Tg ligger under 100 °C. Detta förfarande kan även användas vid Tg-temperaturer över 100 °C, men kräver då speciell utrustning för att t.ex. åstadkomma överhettad ånga.

Ytterligare sätt att värma fibern på kan vara genom IR-värmning, t.ex. genom IR-bestrålning av fiberbanan, alternativt entanglingsvattnet.

Vidare kan annan strålningsuppvärmning utnyttjas samt uppvärmning med mikrovågor. En ytterligare möjlighet är att utnyttja metallviror. Förslagsvis användes en kopparvira, som värmes upp med hjälp av varmluft, varmt vatten eller annan medium eller en kombination av dessa.

Hydroentanglingen kan göras med utgångspunkt från antingen en torrlagd eller en våtlagd fiberbana. Vid torrformning luftlägges torra fibrer på en vira, varefter fiberbanan utsätts för hydroentangling. Vid våtläggning framställes en våt– eller skumformad fiberbana genom att fibrerna dispergeras i vätska alternativt i en uppskummad vätska innehållande en skumbildande tensid och vatten. Ett exempel på ett lämpligt sådant skumformningsförfarande finns i SE 9402470-0. Fiberdispersionen avvattnas på en vira och hydroentanglas därefter. Hydroentanglingen kan ske med konventionell utrustning.

Hydroentanglingen av en våt- eller skumformad fiberbana kan antingen ske in-line, dvs. i direkt anslutning till att fiberbanan avvattnats på viran, eller på ett

våtformat ark som torkats och rullats upp efter formningen. Flera sådana ark kan lamineras samman genom hydroentangling. Det är även möjligt att kombinera torrformning med våt- eller skumformning, på ett sådant sätt att en luftlagd bana av t. ex. syntetfibrer entanglas samman med ett våt- eller skumformat pappersark av massafibrer.

Efter hydroentanglingen pressas och torkas materialet samt rullas upp. Det färdiga materialet kan sedan konverteras på känt sätt till lämpligt format och förpackas.

Enligt en utföringsform av föreliggande förfarande bildas en fiberdispersion av den eller de önskade polymerfibrerna. Fiberdispersionen bildas på en roterande bärare, t. ex. en vira och när dispersionen väl har bildats utsätts den för en hydroentangling genom vattenstrålar som slår mot lagret av fiberdispersionen och på så sätt entanglar eller trasslar ihop fibrerna. Åtminstone i själva hydroentanglingsögonblicket ges sedan polymerfibern en temperatur som överstiger Tg för polymerfibern, men samtidigt understiger dess smältpunkt. Detta kan ske genom att det vatten som utnyttjas för att åstadkomma hydroentanglingen, åtminstone under själva hydroentanglingen, är uppvärmt till en temperatur över Tg för polymerfibern enligt något av ovan angivna sätt. Exempel på energinivåer som utnyttjas är 300 – 600 kWh/ton med ett vattentryck av 80 –120 bar.

Företrädesvis utnyttjas föreliggande förfarande för polymerfibrer med ett Tg ≥ 20 °C, speciellt 20 – 100 °C, lämpligen 50 – 100 °C och mera speciellt 50-70 °C. Tg ligger föäreträdesvis under 150 °C. En speciellt föredragen polymerfiber är polylaktid (PLA) som har ett Tg av 50-70 °C.

Rapporterad glasomvandlingstemperatur för en polymer kan variera mycket dels beroende på att glasomvandlingen sker över ett temperaturintervall och inte vid en viss temperatur, dels beroende på vilken metod som användes för att bestämma glasomvandlingstemperaturen.

En metod som är användbar enligt föreliggande uppfinning för att bestämma glasomvandlingstemperaturen är DSC (Differential Scanning Calorimetry), som mäter förändringen i entalpi som funktion av temperatur. Vid glasomvandlingstemperaturen gör entalpi-temperaturkurvan ett språng och värdet vid detta språng ger glasomvandlingstemperaturen.

En annan metod som anses mera känslig är DMA (Dynamic Mechanical Analysis). I denna metod uppmätes lagringsmodul, förlustmodul, samt tanδ vid en frekvens (normalt 1Hz) som funktion av temperaturen. Vid glasomvandlingstemperaturen ändras lagringsmodulen för en amorf polymer med flera tiopotenser emedan förlustmodulen och tanδ går igenom ett maxima. Med denna metod är det också möjligt att bilda sig en uppfattning om hur mycket modulen ändras vid glasomvandlingstemperaturen. De flesta polymerers glasomvandlingstemperaturer

finns angivna i bl. a för fackmannen välkända handböcker. Tg kan enligt uppfinningen tas fram ur "Polymer Handbook", författare J. Brandrup och E.H. Immergut, förlag "Interscience publishers". Tg kan också tas fram med hjälp av någon av metoderna DSC eller DMA.

Förfarandet enligt uppfinningen är speciellt lämpat för fibrer med hög böjstyvhet. Hög böjstyvhet kan uppnås antingen genom ett högt E-modulvärde eller en grov fibertjocklek. Detta innebär således att speciellt lämpade polymerfibrer är antingen sådana med högt E-modulvärde eller polymerfibrer med mycket grova fibrer. T. ex. en tunn fiber med högt E-modulvärde eller en tjock fiber med mera måttligt eller lågt E-modulvärde. Alternativt kan även sådana där både E-modulvärde och tjocklek är höga utnyttjas. E-modulvärdet för en polymerfiber uttryckes i cN/tex.

Mätning av E-modulvärdet för en fiber kan t.ex. göras genom mätning av den initiala lutningen i ett spännings-töjningsdiagram från en dragprovningstest; utfört enligt svensk standard SS-EN ISO 5079. Ett exempel på utrustning som kan utnyttjas för att mäta E-modulen enligt föreliggande uppfinning är en Lenzig Vibrodyn. Med hjälp av DMA är det även möjligt att få en uppfattning om hur mycket modulen ändras vid glasomvandlingstemperaturen. E-modulvärdet för polymerfibern är, enligt föreliggande uppfinning, E-modulvärdet för fibern vid rumstemperatur (se SS-EN ISO 5079).

Fibrer av alla tjocklekar kan utnyttjas, dvs. såväl mikrofibrer, normaltjocka kring 1-2 dtex, som tjocka kring 6-7 dtex. Enligt en speciell utföringsform kan mycket grova fibrer entanglas till ett fibertyg med hög bulk.

Enligt uppfinningen åstadkommes också nya material, dvs. nya fibertyger som framställts genom hydroentangling.

Lämpligen har polymerfibern ett E-modulvärde av ≥ 20 cN/tex, speciellt ≥50 cN/tex och ännu hellre ≥100 cN/tex. Speciellt kan fibertyger av polymerer med väldigt höga E-modulvärden som 100-2000 cN/tex, speciellt 500-1500 cN/tex, mera speciellt 200-750 cN/tex och ännu mera speciellt 250-600 cN/tex åstadkommas.

Enligt en utföringsform kan förfarandet enligt uppfinningen utnyttjas för framtällningen av mycket starka fibertyg av fibrer med mycket höga E-modulvärden, t.ex. aromatiska polyamider och aromatiska polyestrar.

Ytterligare av speciellt intresse är att enligt uppfinningen tillverka fibertyger med hög bulkmassa. Med hjälp av förfarandet kan fibertyger med mycket tjocka fibrer, t. ex. 6-7 dtex, tillverkas, vilket kan ge ett fibertyg med mycket hög bulkmassa.

Med grova fibrer avses generellt fibrer ≥ 5 dtex och med hjälp av sådana fibrer kan ett material med mycket hög bulk ≥ 8 cm^3/g åstadkommas enligt föreliggande uppfinning. Bulk uttryckes som tjocklek/ytvikt på materialet (cm^3/g).

Enligt föreliggande uppfinning åstadkommes t. ex. fibertyger med en bulk av 5-15 cm^3/g, speciellt 8-15 cm^3/g och mera speciellt 10-15 cm^3/g.

Exempel på ett fibertyg framställt med hjälp av föreliggande uppfinning är ett fibertyg med en mycket hög bulk av 10-15 cm^3/g, en produkt som har mycket goda återfjädringsegenskaper. Härvid användes fibrer med en dimension av 25-50 µm. Sådana fibrer är p.g.a. sin styvhet mycket svåra att entangla på annat sätt. Ett sådant material är speciellt användbart som spridningskikt i blöjor, men kan användas inom många andra områden där hög bulk och goda återfjädringsegenskaper är önskade egenskaper, t.ex. som torkduk.

Speciellt föredraget är att kunna framställa material av semi-kristallina polymerer, med fibrer av tjock diameter och /eller högt E-modulvärde.

Det fibertyg som framställes kan, genom det mindre beroendet av fiberns styvhet, ge ett fibertyg som till huvudsakligen 100 % består av polymerfibern eller fiberblandningen. Dvs. en fiber som inte behöver tillföras mjukgörare eller andra tillsatser som annars skulle krävas för att hantera t. ex. en styv fiber.

Föreliggande förfarande för framställning av fibertyg innebär således ett sätt som är mindre beroende än tidigare sätt av fiberns böjstyvhet och ger, som framgår ovan, olika möjligheter att utnyttja den lediga potential som skapas. Nya material med nya egenskaper kan framställas. T. ex. kan en fiber sträckas optimalt innan hydroentanglingen så att den så styv som möjligt och entanglas. Exempel på lämpliga sådana fibrer är polyesterfibrer och polypropylenfibrer. Genom sträckning kan en fibers brottstyrka ökas för att ge fibern och det därav framställda fibertyget nya egenskaper och med hjälp av föreliggande förfarande kan en sådan fiber hydroentanglas. En på så sätt förbehandlad fiber är ofta inte möjlig att hydroentangla med dagens kända metoder.

Innebörden av uppfinningen är att fibrer med högre styvhet och/eller högre grovlek än vad som normalt användes vid hydroentangling kan entanglas till hög entanglingsgrad vid rimliga energinivåer. Alternativt kan fibrer med, för hydroentangling, normal styvhet och grovlek entanglas vid lägre energinivå eller till en högre entanglingsgrad.

Förfarandet innebär att fibertyg innehållande mycket grova fibrer med lätthet låter sig entanglas och därvid åstadkommes ett material med hög bulk och goda återfjädringsegenskaper.

En ytterligare fördel är att material till en lägre kostnad kan tillverkas, då tillverkningskostnaden för syntetiska fibrer är dimensionsrelaterad och sjunker med ökad fibergrovlek.

Ännu en fördel är att fibrer med mycket hög styrka kan entanglas till ett fibertyg med mycket goda mekaniska egenskaper, framförallt hög våtstyrka, utan att

dessa fibrers samtidigt höga böjstyvhet påverkar entanglingsgraden eller energiinsatsen i negativ bemärkelse.

Sammanfattningsvis kan sägas att föreliggande uppfinning skapar en potential, inte bara genom att vidga antalet aktuella fiberslag avseende polymer och dimension, utan dessutom genom möjligheten att optimera de ingående fiberkomponenterna utifrån andra kriterier än att begränsa fiberns böjstyvhet.

Denna potential kan utnyttjas till förbättrade materialegenskaper (ökad bulk, återfjädring, dragstyrka m.m.) eller sänkta kostnader i form av lägre energiförbrukning eller lägre kostnader för ingående komponenter.

Som nämnts tidigare kan fibertyget omfatta olika blandningar av fibrer, däribland blandningar med icke-syntetiska fibrer. Ju större andel syntetiska polymerfibrer som får ingå , desto större möjlighet finns att utnyttja den fria potential som uppnås. Fibertyget får naturligtvis också väldigt olika egenskaper beroende på inblandningsgrad och typ av fiber. Sammantaget skapas genom föreliggande uppfinning stora möjligheter till optimering samt nya material.

Fibertyget framställt enligt föreliggande uppfinning kan användas bl. a. som avtorkningsmaterial för hushållsbruk eller för industriellt bruk, såsom för storförbrukare som verkstäder, industrier, sjukhus och andra offentliga inrättningar. Det är även användbart som engångsmaterial inom sjukvård, t. ex. som operationsrockar, lakan och liknande. Vidare kan det användas för hygienbruk, t. ex. som komponent i absorptionsprodukter som bindor, trosskydd, blöjor, inkontinensprodukter, sängunderlägg, sårförband, kompresser och liknande. I synnerhet gäller detta fibertyg framställda enligt föreliggande uppfinning med hög våtstyrka. Fibertyg med hög bulkmassa är speciellt fördelaktiga att användas som t.ex. spridningskikt i blöjor, men även som avtorkningsmaterial för hushållsbruk.

Av exempel 1, nedan, framgår att tensidstyrkan för fibern (PLA-fibern) har ökats 20-25 % genom att Tg-temperaturen har överskridits vid hydroentanglingen. Detta ger således en potential av 20-25 % som kan utnyttjas på olika sätt. Såsom framgår av exempel 1 kan ett starkare material erhållas, men potentialen kan också utnyttjas för att ge en energibesparing och därmed en kostnadsbesparing.

EXEMPEL 1

En skumformad fiberdispersion bestående av 60 % massafiber av kemisk sulfatmassa samt 40 % termoplastisk syntetfiber (1,7 dtex, 19 mm) bildades på en roterande vira. Fiberdispersionen hydroentanglades från en sida vid en energiinsats av 300kWh/ton.

Försöket omfattar 3 olika varianter (försök 1, 2 och 3) av polylaktidfiber (PLA, med Tg = 50-70 °C), samt polyamid 6 (PA, med Tg = 50 °C; försök 5) som

termoplastisk syntetfiber. Vid varje försök utfördes hydroentanglingen dels med rumstempererat vatten (20 °C), dels med vatten som värmts till 75 °C.

Som jämförelse gjordes ett försök (försök 4) enligt samma utförande med en fiber av polyetylentereftalat (Tg = 85 °C).

Dragstyrkan i torrt och vått tillstånd (vatten- och tensidlösning) liksom töjning, ytvikt, bulk, etc. mättes och värdena redovisas i tabell 1 nedan.

Tg uppmättes med hjälp av en Perkin Elmer DSC 7 och mätningen utfördes från rumstemperatur till 50 °C över smältpunkten.

E-modulvärdena erhölls på följande vis. Dragprovning gjordes på en Lenzig Vibrodyn med draghastigheten 50 mm/min och inspänningslängden 10 mm. En vikt på 100 mg användes för att förspänna fibern. E-modulen beräknades manuellt genom att tangenten till dragprovningskurvorna i det linjära området ritades in. De i tabellen angivna värdena är E-modulvärdena vid rumstemperatur.

Tabell 1

Försök nr	Polymer-fiber	Temperatur (°C)	Ytvikt (g/m^2)	Tjocklek (µm)	Bulk (cm^3/g)	Dragstyvhets-index (Nm/g)	Dragindex torr (Nm/g)	Töjningsindex (%)	Brottarbets-index (J/g)	Dragindex vatten (Nm/g)	Dragindex tensid (Nm/g)	E-modul (cN/tex)
1	PLA	20	87.7	474	5.4	114	14	74	5.8	12.1	10	210
	PLA	75	91.8	408	4.4	100	14	57	6	12.3	12.1	210
2	PLA	20	91	521	5.7	73	14	44	4.3	8.8	7.4	
	PLA	75	89.8	493	5.5	65	14	47	4.5	9.3	9.1	
3	PLA	20	85.6	490	5.7	71.1	19.9	52	7.2	13.9	11.1	502
	PLA	75	91.3	487	5.3	89.7	19.2	56	7.3	15.4	13.4	502
4	PET	20	85.3	499	5.8	51.6	23.5	66	8.7	20.3	11.8	
	PET	75	85.6	476	5.6	55	23.5	62	8.5	20.8	13.2	
5	PA	20	90.8	503	5.5	78.4	25.6	81.3	11.9	14.1	5.6	
5	PA	75	88.1	466	5.3	123.4	30.0	75.6	13.1	18.1	8.9	

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I torrt tillstånd har fibrerna en relativt hög friktion gentemot varandra och torrstyrkan i fibertyget beror i hög grad av de individuella fibrernas mekaniska egenskaper som hållfasthet, töjning och E-modul.

Som framgår av tabell 1 påverkas styrkan i torrt tillstånd i stort sätt inte alls eller relativt lite, vilket indikerar att fibrerna återfått sina ursprungliga mekaniska egenskaper efter den momentana värmebehandling de utsatts för.

Då dragprov utföres i vatten kommer fibrerna att glida lättare mot varandra varvid graden av mekanisk bindning (intrassling) ökar något i betydelse för fibertygets mekaniska egenskaper. Tabell 1 visar att för samtliga prover är dragindex något högre för de material som är hydroentanglade med varmt vatten.

Vid dragprovning i tensidlösning upphävs i stort sett friktionen mellan fibrerna, vilket gör att graden av intrassling kommer att dominera som påverkande faktor för fibertygets mekaniska egenskaper. Som framgår av tabell 1 ser vi här en tydlig ökning av mellan 20 och 25 % av dragindex för PLA och över 50 % för PA då materialet hydroentanglats i varmt vatten.

Värdena på styvhetsindex som i stort sett är oförändrade (vissa har ökat något, vissa har sjunkit något) visar att bindningstypen är oförändrad. Om det vore så att ökningen i tensidstyrka beror på att fibrerna blivit termiskt bundna till varandra vid värmebehandlingen skulle detta även ha visat sig i en dramatisk ökning av styvhetsindex. För att uppnå en termisk bindning med polylaktid krävs dock betydligt högre temperaturer.

Vad som åstadkommits är således ett fibertyg där fibrerna har sina ursprungliga mekaniska värden, men där strukturen har ändrats på så sätt att fibrerna nått en högre grad av intrassling.

PATENTKRAV

1. Förfarande för hydroentangling av polymerfibrer för framställning av ett fibertyg, **kännetecknat av** att polymerfibern, i hydroentanglingsögonblicket, ges en temperatur som är lika med eller överstiger glasomvandlingstemperaturen (Tg) för polymerfibern samt understiger smälttemperaturen för polymerfibern.

2. Förfarande enligt krav 1, **kännetecknat av** att polymerfibern har en E-modul ≥ 50 cN/tex, vid rumstemperatur.

3. Förfarande enligt krav 1, **kännetecknat av** att polymerfibern har en E-modul ≥ 100 cN/tex, vid rumstemperatur.

4. Förfarande enligt krav 3, **kännetecknat av** att polymerfibern har en E-modul av 100 – 2000 cN/tex, speciellt 500-1500 cN/tex, mera speciellt 200-750 cN/tex och ännu mera speciellt 250-600 cN/tex, vid rumstemperatur.

5. Förfarande enligt något av kraven 1-4, **kännetecknat av** att temperaturen åstadkommes med hjälp av varmt eller överhettat vatten.

6. Förfarande enligt något av kraven 1-4, **kännetecknat av** att temperaturen åstadkommes med hjälp av IR-värme.

7. Förfarande enligt något av kraven 1-4, **kännetecknat av** att temperaturen åstadkommes med hjälp av mikrovågor.

8. Förfarande enligt något av kraven 1-7, **kännetecknat av** att polymerfibern har en glasomvandlingstemperatur (Tg) av ≥ 20 °C.

9. Förfarande enligt något av kraven 8, **kännetecknat av** att polymerfibern har en glasomvandlingstemperatur (Tg) av 20 – 100 °C, speciellt 50-70 °C.

10. Förfarande enligt något av kraven 1-9, **kännetecknat av** att den i polymerfibrerna ingående polymeren innefattar polyester, polylaktid, polyamid eller polypropen, eller sampolymerer eller blandningar därav.

11. Hydroentanglat fibertyg innefattande polymerfibrer, **kännetecknat av** att polymerfibrerna i fibertyget har en E-modul ≥ 50 cN/tex, vid rumstemperatur.

12. Fibertyg enligt krav 10, **kännetecknat av** att polymerfibrerna i fibertyget har en E-modul av 100-2000 cN/tex, speciellt 500-1500 cN/tex, mera speciellt 200-750 cN/tex och ännu mera speciellt 250-600 cN/tex, vid rumstemperatur.

13. Fibertyg enligt något av kraven 10-11, **kännetecknat av** att polymerfibrerna i fibertyget har en glasomvandlingstemperatur (Tg) av ≥ 20 °C.

14. Fibertyg enligt krav 12, **kännetecknat av** att polymerfibrerna i fibertyget har en glasomvandlingstemperatur (Tg) av 20-100 °C, speciellt 50-70 °C.

15. Fibertyg enligt något av kraven 10-13, **kännetecknat av** att fibertyget har en bulkmassa av ≥ 8 cm^3/g.

16. Fibertyg enligt krav 14, **kännetecknat av** att fibertyget har en bulkmassa av 8 – 15 cm^3/g, speciellt 10-15 cm^3/g.

17. Fibertyg enligt något av kraven 10-15, **kännetecknat av** att den i polymerfibrerna ingående polymeren innefattar polyester, polylaktid, polyamid eller polypropen, eller sampolymerer eller blandningar därav.

SAMMANDRAG

Uppfinningen avser ett förfarande för bindning av polymerfibrer till ett fibertyg samt fibertyg framställd med hjälp därav. Enligt uppfinningen utsätts polymerfibrerna för en hydroentangling, varvid polymerfibrerna i hydroentanglingsögonblicket ges en temperatur som överstiger polymerfiberns glasomvandlingstemperatur, men understiger dess smälttemperatur.

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(54) Title: FIBER BONDING

(57) Abstract: The invention relates to a method of bonding polymer fibers into a nonwoven fabric and a nonwoven fabric manufactured with the aid thereof. According to the invention, the polymer fibers are subjected to hydroentangling, the polymer fibers at the moment of hydroentangling being imparted a temperature exceeding the glass transition temperature of the polymer fiber, but being less than its melting point.

FIBER BONDING

Technical field

The invention relates to a method for bonding polymer fibers into a nonwoven fabric and a nonwoven fabric manufactured with the aid thereof.

According to the invention the polymer fibers are subjected to hydroentangling, the fibers at the moment of hydroentangling being imparted a temperature exceeding the glass transition temperature of the polymer fiber and being less than the melting point of the polymer fiber.

Background of the invention

The manufacture of cloth and fabrics is very old art. In addition to the wellknown methods of weaving, knitting and crocheting, there are now a number of methods for the manufacture of nonwoven materials. Such materials can be made of both synthetic fibers and natural fibers. A number of methods utilize heat to melt the fibers together, so-called thermobonding.

There are also other bonding methods, such as stitch-bonding and hydroentangling or spun-lacing. Hydroentangling or spun-lacing is a technology which was introduced in the nineteen seventies. The method involves the forming of a fiber web, either dry-laid or wet-laid, whereafter the fibers are entangled together with the aid of very fine jets of water under high pressure. Several rows of water jets are directed towards the fiber web, which is carried by a moving wire or drum. Finally, the entangled fiber web is dried.

In hydroentangled materials many different types and mixtures of fibers are used, such as for example synthetic staple fibers, synthetic continuous fibers, staple fibers

of regenerated cellulose and pulp fibers. Examples of commercially available fibers of regenerated cellulose are rayon, viscose fibers and lyocell.

Hydroentangling is a method of bonding fibers without using bonding agents or bonding fibers. Hydroentangled material or spun-lace material of high quality can be made at reasonable cost, and has a high absorption capacity, good mechanical properties and a high textile comfort. They were used as wiping materials for house-hold or industrial use, as disposable materials in medical contexts and for hygiene etc.

In order to form a composite web by hydroentangling, the fibers to be entangled must have the right properties for this purpose.

One of several critical factors is the flexural rigidity of the fiber = E x I, where E is the initial modulus of the fiber or the modulus of elasticity, and I is the moment of inertia. The moment of inertia is a sectional quantity ($I = \pi d^4/64$ for a circular cross section) which is dependent on the diameter of the fiber. The initial modulus is thus a material parameter, which is temperature-dependent.

A stiff fiber is more difficult to entangle and requires more specific energy (kWh/ton) to be bonded than a soft fiber, and this in turn limits the range of fibers which are technically and commercially of interest for use in this technology.

Fibers made of thermoplastic polymers, such as e.g. polypropylene, polyester, polyamide commonly occur in hydroentangling.

The properties of thermoplastic and other synthetic fibers are dependent on the properties of the component of polymer or polymers and the type of process used. Often the properties of the polymer cannot be fully utilized. Rather, a compromise must be made due to the technical constraints of the process.

It is typically very difficult to manufacture a fiber which is both very strong and at the same time has a low initial modulus. The strength of the fiber is largely determined by the orientation, imodulus follow the same tendency, i.e. a high fiber strength is accompanied by a high initial modulus.

CA 841 938 describes the manufacture of a nonwoven fabric by hydroentangling, whereby water under high pressure is pressed through a perforated carrier against a sheet of a fiber suspension, to give rise to entangling of the fibers.

WO 95/06769 describes a method and an apparatus for achieving a melt bond and possibly entangling of fibers in a fiber fabric, e.g. a nonwoven fabric. In this case, a steam jet or a jet of superheated steam is used to both melt and entangle the fibers. If one uses jets such as are normally used in hydroentangling, they must be sufficiently hot to melt a meltable component incorporated in the fiber web. WO95/06769 thus describes a process whereby a certain amount of melting of the meltable component is always achieved. This meltable component can either be the fibers themselves or an added meltable component in the form of a powder or granulate. No process for pure hydroentangling is described.

US 3 322 584 describes a melt-bonding process for bonding together two plastic webs. The method described can also be used to bond together two layers of plastic fibers, but this also refers to a melt bond, the temperature used being sufficiently high to melt the fibers.

US 5 069 735 describes a method of edge-melting adjacent sheets or fabrics to solve the problem that these sheets normally flock and are unsuitable for use in operations for example.

US 3 192 560 describes a method for controlled melt-bonding of fiber threads with a suitable medium, e.g. steam or superheated steam, the temperature being kept about or slightly under the melting point of the fiber threads.

One problem with hydroentangling is that the fiber components used must have a flexural rigidity such that the fibers can be entangled using reasonable energy levels. This means a limitation as regards the types of fibers used and means that thin fibers or fibers with a low initial modulus must be used, even if the fibers per se are not optimal for the formation of the fibrous web or for the functional properties of the finished material.

One purpose of the present invention is to achieve a process for manufacturing a nonwoven fabric by hydroentangling, where the flexural rigidity of the fiber components used does not limit the degree of entangling as much as previously.

An additional purpose of the present invention is to achieve a process making it possible to manufacture nonwoven fabrics having coarser fibers, consuming less energy or being stronger than what is possible with today's methods.

Another additional purpose of the present invention is to provide a nonwoven fabric with special properties, such as good mechanical properties, high bulk, etc.

Summary of the invention

We have now found that by raising the temperature at the very moment of entangling it is possible to reduce the flexural rigidity of the fibers and achieve a higher degree of entangling.

We have also found that it is only at the moment of entanglement that a too high initial modulus is of disadvantage. By reducing the initial modulus only during the entangling itself, and thereafter allowing the initial modulus to return to the original

level, a method and a material are provided having great advantages over those previously known.

There is less need to compromise in the fiber manufacturing process between the properties strength and rigidity. The strength of the fiber can instead be fully optimized.

At the same time it is possible to select fibers for the fabric to be bonded, based on other criteria than the limitations placed by the entangling process. In many cases it is an advantage to have stiff fibers in the finished fabric, depending on to what use the material is to be put.

Fibers already well adapted to hydroentangling can be further optimized, providing improved material properties and/or lower energy consumption in the process.

According to the invention, a process is achieved for hydroentangling of polymer fibers for producing a nonwoven fabric. The polymer fiber is imparted at the moment of hydroentangling, a temperature which is equal to or exceeds the glass transition temperature for the polymer fiber and is below the melting point for the polymer fiber.

According to the invention, there is further achieved a hydroentangled fabric comprising polymer fibers, the polymer fibers in the fabric having an initial modulus ≥ 50 cN/tex .

Furthermore, according to the invention a hydroentangled polymer fiber fabric is obtained with a bulk specific volume of ≥ 8 cm^3/g.

Additional embodiments are disclosed in the accompanying subclaims.

Detailed description of the invention

According to the invention the polymer fiber is heated so that at the moment of entanglement it reaches a temperature above the glass transition temperature (Tg) of the polymer fiber. At this temperature the mobility in the molecules increases to such a degree that the stiffness is dramatically affected and a reduction in the modulus of elasticity or initial modulus by up to several powers of 10 can be obtained.

The mechanical properties of synthetic polymers are changed dramatically at the glass transition temperature of the polymer. By momentarily heating the desired fiber to the glass transition temperature or slightly thereabove at hydroentangling, the flexural rigidity and the degree of entangling in the fiber fabric are increased.

Many different types and mixtures of polymer fibers can be used. Of particular preference according to the invention is a nonwoven fabric which completely or partially comprises synthetic polymer fibers, or mixtures or copolymers of such polymer fibers. Based primarily on the purpose of the nonwoven fabric, the type of fiber and the percentage of natural fiber are selected. The greater the percentage of synthetic polymers included in the nonwoven fabric, the greater are the possibilities.

Examples of fibers which can be used in the material according to the present invention are synthetic staple fibers, synthetic continuous fibers, staple fibers of regenerated cellulose, natural fibers such as plant fibers, pulp fibers or mixtures thereof. Examples of commercially available fibers of regenerated cellulose are rayon, viscose and lyocell. Examples of synthetic fibers are fibers of polyester, polylactic acid , polyamide, polypropylene, polybutylene terephthalat (PBT), polyethylene (PE), polyethylene terephthalat (PET) and copolymers thereof, such as polyesteramides. Bicomponent fibers, i.e. fibers having a core of a first polymer, e.g. PET, and a casing of a second polymer, e.g. PE, may be used. The synthetic polymer fibers can comprise both polymer fibers made of natural fibers and polymer fibers made of

synthetic fibers. Even continuous filaments, such as melt-blown and spun-bond fibers can be used as well as profiled so-called capillary fibers. These profiled fibers are often very stiff and nor-mally difficult to handle, but can be entangled with the aid of the present invention. Even mixtures of these different fibers can be used. A typical mixture is 40-50% long, synthetic fibers and the rest pulp, but all mixtures are applicable. The pulp
fibers can be of chemical, mechanical, thermomechanical, chemomechanical or chemothermomechanical pulp (CTMP). Mixing in of mechanical, thermomechanical, chemomechanical or chemothermomechanical pulp fibers provides a material with higher bulk and improved absorption and softness, which is described in SE 9500585-6.

According to the invention, primarily thermoplastic, synthetic polymers and in particular semi-crystalline polymers can be used. Amorphous polymers can also be used.

The heating of the polymer fiber at the moment of hydroentangling can be done in many different ways. One method of achieving a momentary temperature rise in the process is to heat the entangling water to such a temperature that the fiber, at the moment of hydroentangling reaches a temperature above Tg. Suitably this process is used when the Tg of the polymer lies below 100°C. This process can also be used at Tg-temperatures above 100°C, but will then require special equipment to produce superheated steam.

A further method of heating the fiber can be by IR-heating, e.g. by IR-radiation of the fiber web, or alternatively of the entangling water.

Other radiation heating can be used or microwave heating. A further possibility is to use metal wires, e.g. of copper, which is heated up with the aid of hot air, hot water or another medium or a combination thereof.

The hydroentangling can be done starting from either a dry-laid or a wet-laid fiber web. In dry-forming, the dry fibers are air-laid on a wire, whereafter the fiber web is subjected to hydroentangling. In wet-laying, a wet- or foam-formed web is made by the fibers being dispersed in liquid or in a foam liquid containing a foam-forming tenside and water. An example of a suitable foam-forming process of this type is described in SE 9402470-0. The fiber dispersion can be dewatered on a wire and thereafter be hydroentangled. The hydroentangling can be effected with conventional equipment.

Hydroentangling of a wet- or foam-formed fiber web can be done either in-line, i.e. directly when the fiber web has been dewatered on the wire, or on a wet-formed sheet which has been dried and rolled up after forming. Several such sheets can be laminated together by hydroentangling. It is also possible to combine dry-forming with wet- or foam-forming in such a way that an air-laid web of synthetic fibers, for example, is entangled together with a wet- or foam-formed paper sheet of pulp fibers.

After the hydroentangling, the material is pressed and dried and rolled up. The finished material can then be converted in a known manner to a suitable format and be packed.

According to one embodiment of the present process, a fiber dispersion is formed of the desired polymer fiber(s). The fiber dispersion is formed on a rotating carrier, e.g. a wire, and when the dispersion has been formed it is subjected to hydro-entangling by water jets striking against the layer of the fiber dispersion and in this manner entangling the fibers. At the moment of hydroentangling at least, the polymer fiber is imparted a temperature exceeding Tg for the polymer fiber, but at the same time being less than its melting point. This is done by heating the water used to produce the hydroentangling, at least during the hydroentangling itself, to a temperature

above Tg for the polymer fiber by one of the above ways. Typical energy levels used are 300 – 600 kWh/ton with a water pressure of 80 – 120 bar.

Preferably, the present invention is used for polymer fibers with a Tg ≥ 20°C, especially 20 - 100°C, suitably 50 - 100°C and more particularly 50 - 70°C. Tg is preferably below 150°C. A particularly preferred polymer fiber is polylactic acid (PLA), which has a Tg of 50 - 70°C.

The reported glass transition temperature for a polymer can vary widely, on one hand due to the fact that glass transition occurs over a temperature interval and not at a certain temperature, and on the other hand depending on what method is used to determine the glass transition temperature.

One method which is usable in the present invention to determine the glass transition temperature is DSC (Differential Scanning Calorimetry), which measures the change in enthalpy as a function of temperature. At the glass transition temperature, the enthalpy-temperature curve makes it jump, and the value at this jump gives the glass transition temperature.

Another method which is considered more sensitive is DMA (Dynamic Mechanical Analysis). In this method the storage modulus, the loss modulus and tanδ are measured at a frequency (normally 1 Hz) as a function of temperature. At the glass transition temperature, the storage module for an amorphous polymer changes by several orders of ten, while the loss module and tanδ go through maximums. With this method it is also possible to get an idea of how much the modulus changes at the glass transition temperature. The glass transition temperatures of most polymers are given in handbooks well-known to the person skilled in the art. For the purposes of the invention, Tg can be taken from "Polymer Handbook" by J. Brandrup and E.H. Immergut, publisher "Interscience Publishers". Tg can also be derived with the aid of one of the methods DSC or DMA.

The method according to the invention is particularly suited to fibers with high flexural rigidity. High flexural rigidity can be achieved either by a high initial modulus value or by high fiber thickness. This means that polymer fibers especially suitable are either fibers with a high initial modulus or very thick polymer fibers, for example a thin fiber with a high initial modulus or a thick fiber with a less pronounced or low initial modulus. Alternatively, fibers which are both thick and have a high initial modulus can be used. The initial modulus for a polymer fiber is expressed in cN/tex.

Measurement of the initial modulus value for a fiber can for example be done by measuring the initial slope of a stress-strain diagram from a tensile test performed according to SS-EN ISO 5079. One example of the equipment, which can be used to measure the initial modulus according to the present invention, is a Lenzing Vibrodyn. With the aid of DMA it is also possible to get an idea of how much the modulus changes at the glass transition temperature. The initial modulus value for the polymer fiber is, in accordance with the present invention, the initial modulus value for the fiber at room temperature (see SS-EN ISO 5079).

Fibers of all thicknesses can be used, i.e. both microfibers, fibers of normal thickness about 1-2 dtex, and thick fibers about 6-7 dtex. According to a special embodiment, very thick fibers can be entangled to form a fiber fabric with high bulk.

According to the invention, new materials are also achieved, i.e. new nonwoven fabrics produced by hydroentangling.

Suitably, the polymer fiber has an initial modulus value $\geq$ 20 cN/tex, especially $\geq$ 50 cN/tex and most preferably $\geq$ 100 cN/tex. It is also possible to achieve nonwoven fabrics of polymers with very high initial modulus values, such as 100-2000 cN/tex, especially 500-1500 cN/tex, more particularly 200-750 cN/tex, and even more particularly 250-600 cN/tex.

According to one embodiment, the process according to the invention can be used to produce very strong nonwoven fabrics of fibers with very high initial modulus values, e.g. aromatic polyamides and aromatic polyesters.

Of special interest is the fact that according to the invention it is possible to manufacture nonwoven fabrics having a high bulk. With the aid of the present method, nonwoven fabrics having very thick fibers, e.g. 6-7 dtex, and which provide a nonwoven fabric with very high bulk specific volume, can be manufactured.

Thick fibers generally refer to fibers $\geq$ 5 dtex, but with the aid of such fibers a material with very high bulk specific volume $\geq$ 8 cm^3/g can be achieved in accordance with the present invention. Bulk is expressed as thickness divided by surface weight of the material (cm^3/g).

According to the present invention there are achieved nonwoven fabrics with a bulk specific volume of 5-15 cm^3/g, especially 8-15 cm^3/g and more particularly 10-15 cm^3/g.

One example of a nonwoven fabric manufactured with the aid of the present invention is a nonwoven fabric with a very high bulk of 10-50 cm^3/g, a product, which has very good resilience. 25-50 µm fibers are used in this case. Such fibers, due to their stiffness, are very difficult to entangle by other methods. Such a material is particularly usable as a wicking layer in diapers, but it can be used in a number of different areas, where high bulk and good resilience are desirable properties, such as wipes.

It is of particular advantage to be able to make materials of semi-crystalline polymers, with fibers of thick diameter and/or high modulus of elasticity.

The nonwoven fabric produced, by being less dependent on the stiffness of the fiber, can provide a nonwoven fabric consisting substantially 100% of the polymer fiber or the fiber mixture, i.e. a fiber to which no softeners or other additives need to be added as might be required to handle a stiff fiber, for example.

The present method for manufacturing nonwoven fabrics is thus less dependent than previous methods on the flexural rigidity of the fiber and, as is evident from the above, provide various possibilities to exploit the potential created. New materials with new properties can be made. For example, a fiber can be optimally stretched before hydroentangling so that it is as stiff as possible, and be entangled. Examples of suitable fibers of this type are polyester fibers and polypropylene fibers. By stretching, the breaking strength of these fibers can be increased to impart new properties to the fiber and the nonwoven fabric produced therefrom and with the aid of the present method, such a fiber can be hydroentangled. A fiber pretreated in this manner is often impossible to hydroentangle by today's methods.

The significance of the invention is that fibers having greater stiffness and/or greater thickness than what is normally used in hydroentangling, can be entangled to a high degree of entanglement at reasonable energy levels. Alternatively, fibers of normal stiffness and thickness for hydroentangling can be entangled at lower energy levels and/or to a higher degree of entanglement.

The method allows nonwoven fabrics containing very thick fibers to be easily entangled and thus achieve a material of high bulk and good resilience.

An additional advantage is that material can be manufactured at lower cost since the manufacturing cost of synthetic fibers is dimension-related, decreasing with increasing fiber thickness.

An additional advantage is that fibers of very high strength can be entangled into a nonwoven fabric with very good mechanical properties, primarily high wet strength, without the high flexural rigidity of these fibers negatively affecting the degree of entanglement or the energy consumption.

In conclusion it can be said that the present invention creates a potential, not only by expanding the number of potential types of fibers as regards polymers and dimensions, but also by making it possible to optimize the fiber components used based on other criteria than limiting the flexural rigidity of the fiber.

This potential can be used for improved material properties (increased bulk, resilience, tensile strength, etc.) or reduced costs in the form of lower energy consumption and/or lower cost for the components used.

As was mentioned previously, the nonwoven fabric can comprise various mixtures of fibers, including mixtures of non-synthetic fibers. The greater the percentage of synthetic polymer fibers included, the greater will be the possibility of using the free potential achieved. The nonwoven fabric will, of course, also have many different properties, depending on the degree of admixture and the type of fiber. On the whole, the present invention provides greater possibilities for optimizing and using new materials.

The nonwoven fabric made according to the present invention can be used as i.a. wiping material for household use or for industrial use, such as major consumers including shops, industries, hospitals, etc. It can also be used for disposable hospital articles, e.g. operation coats, sheets and the like. It can also be used for hygienic purposes, e.g. as components in absorbent products such as sanitary napkins, panty liners, diapers, incontinence products, bed protectors, surgical dressings, compresses and the like. This is particularly true of nonwoven fabrics made according to the present invention with high wet strength. Nonwoven fabrics of high bulk specific

volume are particularly advantageous for use as wicking layers in diapers but also as wiping materials for household use.

It is evident from Example 1 below that the tenside strength of the fiber (PLA-fiber) has increased by 20-25% by virtue of the fact that the Tg-temperature was exceeded at hydroentangling. This provides a potential of 20-25%, which can be used in various manners. As can be seen in Example 1, a stronger material can be obtained, but the potential can also be used to provide energy savings and thus savings in costs.

EXAMPLE 1

A foam-formed fiber dispersion consisting of 60% pulp fiber of chemical sulphate pulp and 40% thermoplastic synthetic fiber (1.7 dtex, 19 mm) was formed on a rotating wire. The fiber dispersion was hydroentangled from one side at an ener-gy input of 300 kWh/ton.

The trial encompassed 3 different variants (Trials 1, 2 and 3) of polylactic acid fiber (with Tg = 50-70°C) and polyamide 6 (PA, with Tg = 50 °C; trial 5) as thermoplastic synthetic fiber. At every trial, hydroentangling was performed both with water at room temperature (20°C) and with water heated to 75°C.

As a comparison a trial (trial 4) was made according to the same method with a fiber of polyethylene terephthalat (Tg = 85°C).

The tensile strength in dry and wet states (water and tenside solutions) as well as elongation, surface weight, bulk, etc. were measured and the values are displayed in Table 1 below.

Tg was measured with the aid of a Perkin Elmer DSC 7 and the measurement was done from room temperature to 50°C over the melting point.

The values of initial modulus were obtained in the following manner. The tensile test was performed on a Lenzing Vibrodyn with a pulling speed of 50 mm/min. and a gauge length of 10 mm. A weight of 100 mg was used to pretension the fiber. The initial modulus was calculated manually by drawing in the tangent to the tensile testing curves in the linear range. The values of the initial modulus given in the table are at room temperature.

In the dry state, the fibers have a relatively high friction against each other and the dry strength of the nonwoven fabric largely depends on the mechanical properties of the individual fibers, such as tenacity, elongation and initial modulus.

As can be seen in Table 1, the strength in the dry state is hardly affected at all or to a lesser extent, which indicates that the fibers have reassumed their original mechanical properties after the momentary heat treatment to which they were subjected.

When tensile tests are performed in water, the fibers will slide more easily against each other and the degree of mechanical bonding (entangling) will assume greater importance for the mechanical properties of the nonwoven fabric. Table 1 shows that for all of the trials the tensile index was somewhat greater for those materials which have been hydroentangled with hot water.

For tensile testing in the tenside solution, the friction between the fibers was essentially removed, and this made the degree of entanglement the dominant factor for the mechanical properties of the nonwoven fabric. As is evident from Table 1, there is a significant increase of between 20 and 25% for PLA and more than 50 % for PA in the tensile index when the material was hydroentangled in hot water.

The values of the stiffness index were essentially unchanged (certain of them have increased somewhat, certain have decreased somewhat) and this shows that the type of bonding is unchanged. If the increase in the tenside strength were dependent on

the fact that the fibers had been thermally bonded to each other during the heat treatment, this would also be revealed as a dramatic increase in the rigidity index. In order to achieve thermobonding of polylactic acid, significantly higher temperatures are required however.

What has been achieved is thus a nonwoven fabric where the fibers have their original mechanical values, but where the structure has been changed in such a way that the fibers have been entangled to a higher degree.

Table 1

Trial No.	Poly-mer fiber	Tem pera-ture °C	Surface weight g/m^2	Thick-ness µm	Bulk spe-cific vol-ume cm^3/g	Stiff-ness Index Nm/g	Ten-sile Index Dry Nm/g	Stretch Index %	Work to rupture index J/g	Tensile Index Water Nm/g	Tensile Index Tenside Nm/g	Ini-tial mod-ulus
1	PLA	20	87.7	474	5.4	114	14	74	5.8	12.1	10	210
	PLA	75	91.8	408	4.4	100	14	57	6	12.3	12.1	210
2	PLA	20	91	521	5.7	73	14	44	4.3	8.8	7.4	
	PLA	75	89.8	493	5.5	65	14	47	4.5	9.3	9.1	
3	PLA	20	85.6	490	5.7	71.1	19.9	52	7.2	13.9	11.1	502
	PLA	75	91.3	487	5.3	89.7	19.2	56	7.3	15.4	13.4	502
4	PET	20	85.3	499	5.8	51.6	23.5	66	8.7	20.3	11.8	
	PET	75	85.6	476	5.6	55	23.5	62	8.5	20.8	13.2	
5	PA	20	90.8	503	5.5	78.4	25.6	81.3	11.9	14.1	5.6	
5	PA	75	88.1	466	5.3	123.4	30.0	75.6	13.1	18.1	8.9	

CLAIMS

1. Method of hydroentangling polymer fibers to manufacture a nonwoven fabric, **characterized in** that the polymer fiber, at the moment of hydroentangling, is imparted a temperature equal to or exceeding the glass transition temperature (Tg) of the polymer fiber and being less than the melting point of the polymer fiber.

2. Method according to Claim 1, **characterized in** that the polymer fiber has an initial modulus ≥ 50 cN/tex, at room temperature.

3. Method according to Claim 1, **characterized in** that the polymer fiber has an initial modulus ≥ 100 cN/tex, at room temperature.

4. Method according to Claim 3, **characterized in** that the polymer fiber has an initial modulus of 100 – 2000 cN/tex, especially 500 – 1500 cN/tex, more particularly 200 – 750 cN/tex, and even more particularly 250 – 600 cN/tex, at room temperature.

5. Method according to one of Claims 1 – 4, **characterized in** that the temperature is achieved with the aid of hot or superheated water.

6. Method according to one of Claims 1 – 4, **characterized in** that the temperature is achieved with the aid of IR-heat.

7. Method according to one of Claims 1 – 4, **characterized in** that the temperature is achieved with the aid of microwaves.

8. Method according to one of Claims 1 – 7, **characterized in** that the polymer fiber has a glass transition temperature (Tg) of ≥ 20°C.

9. Method according to one of Claims 1 – 8, **characterized in** that the polymer fiber has a glass transition temperature (Tg) of 20 - 100°C, especially 50 - 70°C.

10. Method according to one of Claims 1 – 9, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid , polyamide or polypropylene, or copolymers or mixtures thereof.

11. Hydroentangled nonwoven fabric comprising polymer fibers, **characterized in** that the polymer fibers in the nonwoven fabric have an initial modulus ≥ 50 cN/tex, at room temperature.

12. Nonwoven fabric according to Claim 10, **characterized in** that the polymer fibers in the nonwoven fabric have an initial modulus of 100 – 2000 cN/tex, especially 500 – 1500 cN/tex, more particularly 200 – 750 cN/tex, and even more particularly 250 – 600 cN/tex, at room temperature.

13. Nonwoven fabric according to one of Claims 10 – 11, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of ≥ 20°C.

14. Nonwoven fabric according to Claim 12, **characterized in** that the polymer fibers in the nonwoven fabric have a glass transition temperature (Tg) of 20 - 100°C, especially 50 - 70°C.

15. Nonwoven fabric according to one of Claims 10 – 13, **characterized in** that the nonwoven fabric has a bulk specific volume of ≥ 8 cm^3/g.

16. Nonwoven fabric according to Claim 14, **characterized in** that the nonwoven fabric has a bulk specific volume of 8 – 15 cm^3/g, especially 10 – 15 cm^3/g.

17. Nonwoven fabric according to one of Claims 10 – 15, **characterized in** that the polymer included in the polymer fibers comprises polyester, polylactic acid , poly-amide or polypropylene, or copolymers or mixtures thereof.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 00/01670

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D04H 1/46
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3485706 A (F.J. EVANS), 23 December 1969 (23.12.69) --	1-17
Y	DE 4013946 A1 (HOECHST AG), 31 October 1991 (31.10.91) --	1-10
Y	US 5286553 A (K. HARAGUCHI ET AL), 15 February 1994 (15.02.94) -- --------	11-17

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
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